

HARVARD COLLEGE

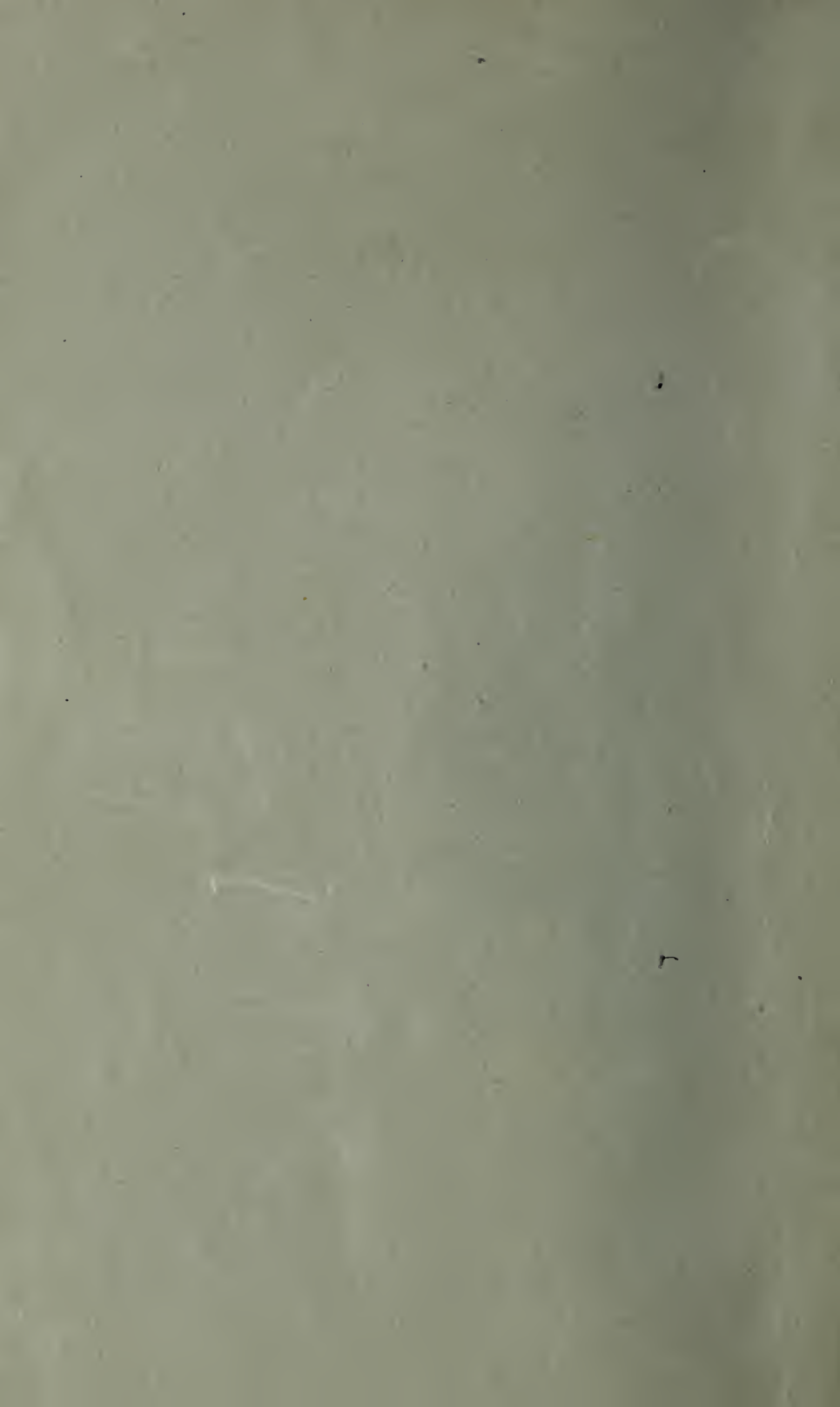


CHEMISTRY 1

INORGANIC CHEMICAL PREPARATIONS

L. CLARKE

1908



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CHEMISTRY 1

INORGANIC PREPARATION II.

PURE SODIUM CHLORIDE.

Common salt contains impurities of sulphates, the alkaline earths, and the so-called heavy metals. These can be eliminated by the following procedure:—

Dissolve 100 grams common salt in 350 cc. water in an evaporating dish. In a beaker, slake about two grams of lime and add sufficient water to make a thin paste, which is known as "milk of lime," add this to the hot salt solution, stir thoroughly and filter. Treat the hot filtrate with barium chloride until all sulphate is precipitated, then add pure sodium carbonate solution in slight excess, which will throw down the alkaline earths. Filter, and add pure hydrochloric acid till neutral. Evaporate to crystallization. Dry the crystals in the air bath.

Write the reactions involved in this purification, and look up the properties of sodium chloride in your lecture note-book.

The pure salt should give a perfectly clear solution, and should give a precipitate neither with barium chloride nor sodium carbonate.

INORGANIC PREPARATION III.

POTASSIUM PERCHLORATE.

In an evaporating dish heat gently 50 grams of potassic chlorate, until, after having melted, it becomes thick and pasty. After cooling, break up the mass and extract twice with 2000 cubic centimetres of cold water to remove potassic chloride; then recrystallize from hot water in the following manner: dissolve the perchlorate in the smallest possible amount of hot water, set aside to cool, and when perfectly cold filter with the Buchner funnel and wash the crystals

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with a few cubic centimetres of cold water. Dry the crystals by exposure to the air on filter paper for a few hours, and transfer to a dry bottle.

Reaction. (a) $4 \text{KClO}_3 = \text{KCl} + 3 \text{KClO}_4$.

(b) $8 \text{KClO}_3 = 5 \text{KClO}_4 + 3 \text{KCl} + 2\text{O}_2$.

Potassic perchlorate is soluble 0.7 parts in 100 of water at 0° ; and 20 parts in 100 of water at 100° .

INORGANIC PREPARATION IV.

CALCIC CHLORIDE (CRYSTALS) $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$.

In an evaporating dish put 100 grams of marble and neutralize with hydrochloric acid (Sp. Gr. 1.2) by adding the acid in small portions at a time, stirring thoroughly and allowing the effervescence practically to stop before adding more acid. When the marble is all dissolved, add enough milk of lime to make the solution slightly alkaline. Add a few cubic centimeters of chlorine water and boil ten or fifteen minutes, then make strongly alkaline with milk of lime, and boil, until a test portion gives no test for iron. (The test for iron is potassium ferrocyanide, $\text{K}_4\text{Fe}(\text{CN})_6$, which gives a blue precipitate in acid solution. Try this with some ferric chloride.) Filter hot, and evaporate the filtrate over the free flame until a crust begins to form around the edge, then set aside to cool slowly.

The crystals of calcic chloride are very deliquescent and so should not be exposed to the air. For a sample, two or three good crystals may be removed from the mother liquor, dried with filter paper hurriedly, and placed in a dessicator over concentrated sulphuric acid a few days, then preserved in a dry, cold, bottle with well-fitting greased stopper.

The mother liquor and remaining crystals can be used for the next preparation.

Calcic chloride prepared in this way should give a clear, neutral solution in water. This solution should not be affected either by hydrogen sulphide or by ammonium sulphide. These reagents detect the presence of the "heavy metals," iron, for example. The aqueous solution of calcic chloride should not give off traces of ammonia when heated with sodic or potassic hydrate.

INORGANIC PREPARATION V.

CALCIC CARBONATE. CaCO_3 .

Add sufficient water to the crystals of calcic chloride obtained in the last preparation, to make a complete solution, and heat nearly to boiling (about 95°). Add a strong filtered solution of ammoniac carbonate to complete precipitation. Allow the precipitate to settle, and pour off the supernatant liquor through a Buchner funnel, finally transferring the precipitate also to the filter, and remove the mother liquor thoroughly by suction. Wash the precipitate with hot water till the last portion of the filtrate is free from chlorides, and dry on filter paper over the hot plate. Bottle the product in a weighed bottle and calculate the yield from the original marble, making allowance for the crystals of the crystallized calcic chloride retained as a sample.

Pure calcic carbonate made in this way dissolves in *pure* dilute hydrochloric acid, giving a clear solution, which should not contain iron, aluminium, magnesium, or appreciable sulphates.

Iron may be detected by potassium ferrocyanide, $\text{K}_4\text{Fe}(\text{CN})_6$. To learn the tests for aluminium and magnesium perform the following test-tube experiments:—

- (1) To aluminic sulphate solution add ammoniac hydrate.
- (2) To magnesian sulphate solution add ammoniac chloride, ammoniac hydrate, and sodic phosphate.

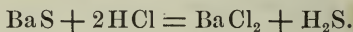
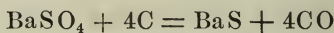
INORGANIC PREPARATION VI.

BARIC CHLORIDE. $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$.

Weigh out 100 grams of barite, or precipitated baric sulphate will do, and add 15 grams of powdered charcoal. Mix very thoroughly and transfer to a clay crucible with a cover. Heat the mixture in the furnace for about an hour and a half, then allow to cool. Extract the crucible contents two or three times with hot water, filter into a flask, fit this with funnel and delivery tubes, and add hydrochloric acid to slight excess. Since a large amount of hydrogen sulphide is evolved this must be done under the hood, and the hydrogen sul-

phide absorbed by caustic soda solution. The tube leading into the flask containing the caustic should not dip below the surface of the liquid but should remain about an eighth of an inch above it. Filter the solution of baric chloride so obtained and evaporate to dryness; redissolve in hot water, filter hot (plaited filter), and evaporate to crystallization. Filter with the Buchner funnel. Recrystallize from distilled water, filter again by suction and dry the crystals in the desiccator. Weigh and calculate the per cent. of yield of baric chloride from the given amount of baric sulphate.

Reactions for the process : —



Read up the account of baric chloride in your lecture notes.

INORGANIC PREPARATION VII.

ZINC CARBONATE ZnCO_3 .

Dissolve 50 grams of zinc sulphate in 50 grams of boiling water and pour slowly with stirring into a boiling solution of 40 grams of sodium bicarbonate in 200 cubic centimetres of water. Allow the precipitate to settle and wash by decantation with boiling water until the washings are free from sulphate. Filter on cloth and dry in the air bath.

This procedure gives essentially pure ZnCO_3 . If normal sodium carbonate were used the precipitate would be a basic salt of indefinite composition. If the sodium bicarbonate were added to the zinc sulphate, a basic carbonate of zinc would be produced, which further quantities of NaHCO_3 would decompose only with much difficulty. It is necessary to have always present an excess of NaHCO_3 .

Zinc carbonate is a pure white powder. Try its solubility in water, NaOH and HCl .

INORGANIC PREPARATION VIII.

ALUMINIC CHLORIDE AlCl_3 .

(Anhydrous.)

Dry aluminic chloride cannot be obtained pure by a wet method, so the following special procedure has to be employed in its preparation.

Fit a 500 cubic centimetre flask with a 2 hole rubber stopper; through one hole of the stopper pass a funnel tube reaching nearly to the bottom of the flask, and through the other, a delivery tube bent at a right angle and connecting with two special wash bottles. (See diagram posted on blackboard in Laboratory.) In the first wash bottle put about 50 cubic centimetres of water, in order to absorb any hydrochloric acid gas which may come over in the subsequent procedure; in the second put the same amount of concentrated sulphuric acid, to absorb moisture from the chlorine gas, which is to be generated later. To the second wash bottle attach a piece of hard glass tubing about 50 centimetres long and 15 millimetres in diameter by means of a glass tube and cork. The other end of the large glass tube is fitted with a cork having a small glass tube about a foot long and pointed up the hood.

Weigh out about 3 grams of aluminium scrap, wash with ether (*beware of flame!*) to remove oil and dry carefully and place in the end of the hard glass tube next the wash bottles.

Fill the flask about one third full of lumps of pyrolusite (black oxide of manganese) and connect up the apparatus, making sure that all the joints are perfectly tight. Add pure concentrated hydrochloric acid enough to cover the pyrolusite and warm gently with the smoky flame so that chlorine is evolved. Pass the chlorine through the apparatus until all the air is driven out, and then with a Bunsen burner heat the aluminium scrap. This soon becomes red hot and an active reaction takes place, while the aluminic chloride formed sublimes over in the cool end of the tube and collects as a yellow solid. The heating with the Bunsen burner is continued until all the aluminium is used up and a small black mass remains; then the burner is taken away and the tube allowed to cool, but the stream of chlorine is kept passing through until the tube is cold enough to be grasped by the hand. Stop the stream of gas by filling up the gas flask with

water and disconnecting from the wash bottles. (Save this liquor for a future experiment.) Disconnect also the hard glass tube containing the sublimate of aluminic chloride. Transfer the AlCl_3 to a weighed *dry* bottle as hurriedly as possible, and weigh. Calculate the per cent of yield of AlCl_3 from the known amount of Al.

As aluminic chloride is very deliquescent, in handling it moisture must be avoided as much as possible. This salt decomposes somewhat on standing and gaseous HCl is set free; therefore be very careful about opening a bottle of this substance, especially after it has been standing for several days.

Throw a small lump of AlCl_3 into a beaker of water; test the solution with litmus paper.

Prepare AlCl_3 in the wet way. How can you do this? Compare it with AlCl_3 prepared by the dry method.

INORGANIC PREPARATION IX.

POTASSIC DICHROMATE $\text{K}_2\text{Cr}_2\text{O}_7$.

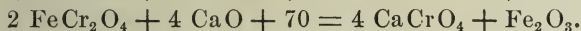
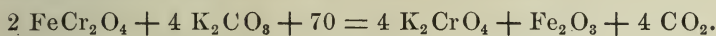
(*From Chrome Iron Ore.*)

Dissolve 56 grams of *pure* K_2CO_3 in 60 grams of water, add 175 grams of lime, and let the mixture stand till the lime has slaked. Evaporate to dryness on the water bath, then heat gently over the free flame with constant stirring until the mass has become a dry powder. Add 112 grams of powdered chromite (chrome iron ore, FeCr_2O_4) and mix thoroughly. Transfer to a clay crucible without cover and heat to a red heat with the oxidizing flame for about 3 hours — stirring occasionally.

When the ignition is over and the mass is cold break up into small lumps and extract with 300 to 400 cubic centimetres of water. Filter, and concentrate the filtrate until crystals begin to appear, at which point add a concentrated solution of 20 grams of K_2SO_4 and filter. To the yellow filtrate add dilute sulphuric acid till strongly acid, and allow the solution to cool. (Make this dilute acid by adding slowly 1 volume of conc. H_2SO_4 to 2 volumes of water contained in a flask. During the addition of the acid to the water, shake the flask and cool under the water tap.) The $\text{K}_2\text{Cr}_2\text{O}_7$ will precipitate partly on the addition of the acid, and more on cooling. When cold, filter, using an asbestos filter and the Buchner funnel. Recrystallize the precipi-

tate by dissolving it in the smallest possible amount of hot water, and allowing it to cool slowly. Filter without crushing the crystals and dry in the drying oven. The first mother liquor contains more of the dichromate but it is so mixed with potassic sulphate that the separation of the two is not practical.

Reactions: The ignition of the potassic carbonate, lime, and chromite mixture forms potassic chromate, calcic chromate, and iron oxide, the oxygen necessary coming from the air. The excess of lime is to keep the mass porous. Any impurity of sand in the chromite forms a slag with the K_2CO_3 . Reactions:—



On treatment of the fused mass with water the chromates go into solution, leaving the iron oxide undissolved. The potassic sulphate decomposes the calcic chromate.



The sulphuric acid changes the potassic chromate into potassic dichromate. Write the reaction.

INORGANIC PREPARATION X.

CHROMIC OXIDE Cr_2O_3 .

Please perform this experiment under the hood.

12.5 grams of sodium dichromate, $\text{Na}_2\text{Cr}_2\text{O}_7$, are moistened with 10 drops of water, and 2 cubic centimeters of glycerine are added, the mixture being in a copper crucible of 500 cubic centimeters capacity. Stir thoroughly, warm on the water bath, and, as soon as the mass *takes fire*, cover the crucible with an earthenware plate. (If the mixture does not take fire of itself after a few moments, set it off with a lighted match held in a pair of tongs.)

When the reaction is complete and the vessel is cold, the resulting green chromic oxide is washed, first with cold, then with hot water, and filtered from all soluble material by means of a plaited filter. Dry the Cr_2O_3 on the water bath and ignite a few moments in a porcelain crucible with the Bunsen lamp. The yield should be about 5 grams.

The reactions taking place between $\text{Na}_2\text{Cr}_2\text{O}_7$ and glycerine $\text{C}_3\text{H}_8\text{O}_3$ are very complex. Two of the products are Cr_2O_3 and Na_2CO_3 , and these will form the major part of the mixture in the crucible after the combustion is over. The treatment with water removes the Na_2CO_3 and also any unchanged $\text{Na}_2\text{Cr}_2\text{O}_7$. The final ignition of the Cr_2O_3 in the porcelain crucible is to destroy any last traces of organic matter.

Try the solubility of a small amount of Cr_2O_3 in water, dilute HCl and NaOH .

INORGANIC PREPARATION XI.

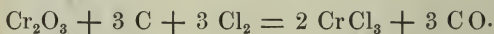
CHROMIC CHLORIDE CrCl_3 .

(*Anhydrous.*)

Dry chromic chloride, like aluminic chloride, cannot be prepared pure in the wet way, so the following method is used:—

Set up an apparatus exactly like that used in the preparation of aluminic chloride (Inorganic Preparation VII), consisting of chlorine generator; two wash bottles, one containing water, the other concentrated sulphuric acid; and the hard glass tube. In the hard glass tube at the end nearest the second wash bottle, place a mixture of 5 grams of Cr_2O_3 and 1.2 grams of charcoal. Pass chlorine gas slowly through the apparatus and bring the end of the hard glass tube containing the oxide-charcoal mixture to a full red heat. The chromic chloride sublimes in the cool end of the tube in shining violet leaflets. When the reaction is complete, allow the tube to cool. When cold stop the current of chlorine, disjoint the hard glass tube, and transfer the CrCl_3 to a dry weighed bottle. Calculate the per cent. yield of CrCl_3 from the weight of Cr_2O_3 taken at the beginning of the experiment.

Reaction: This is called a concurrent reaction. The formation of the CrCl_3 is brought about by the combined action of the C and Cl_2 on the Cr_2O_3 .



Try the solubility of CrCl_3 in pure water and in water containing a small amount of CrCl_3 prepared by the wet method.

Save the liquor in the chlorine generator for the next preparation.

INORGANIC PREPARATION XII.

MANGANOUS CHLORIDE $\text{MnCl}_2 \cdot 4 \text{H}_2\text{O}$.

The source of the manganous chloride is the waste acid liquor from the preparation of chlorine.



Evaporate the liquor to dryness on the steam bath in order to drive off the excess of acid. Add a volume of water equal to that of the liquor used, boil about ten minutes, and filter with a plaited filter.

Measure out *one tenth* of the filtrate and to it add sodium carbonate solution to complete precipitation of the manganese as carbonate and of the impurity of iron as hydrate. Wash several times by decantation, then put this precipitate into the main portion of the above mentioned filtrate and boil gently for half an hour, or until a small test portion treated with ammonium sulphide gives a *flesh colored* precipitate which is soluble in dilute acetic acid. Filter and evaporate to the point of crystallization. The crystals of manganous chloride contain 4 molecules of water — $\text{MnCl}_2 \cdot 4 \text{H}_2\text{O}$ — and are deliquescent.

In the purification of this substance, the addition of MnCO_3 to the solution throws down the impurity of iron as hydrate, while a corresponding quantity of MnCl_2 goes into solution. The amount of iron salt is usually so small that one tenth of the total amount of manganese will give MnCO_3 enough for the purpose. Complete and balance the reaction: —



Divide a solution of a crystal of MnCl_2 in a test tube into two parts: To the first add a few drops of $(\text{NH}_4)_2\text{S}$; to the second a few drops of a solution of Na_2CO_3 .

INORGANIC PREPARATION XIII.

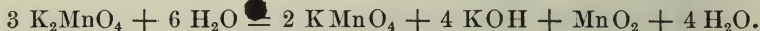
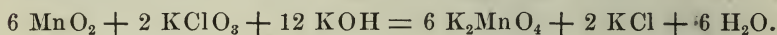
POTASSIC PERMANGANATE KMnO_4 .

Dissolve 35 grams potassic hydroxide in 50 grams of water in an evaporating dish, add 18 grams potassic chlorate, and heat till completely dissolved. Add to this solution, with stirring, 30 grams

of pyrolusite which has previously been ignited on an iron plate to burn out organic matter, and evaporate to a thick paste. This paste is now transferred to a loosely covered iron crucible, dried slowly, and heated to redness over the free flame. The paste turns to a dark green or brown solid mass and water vapor escapes. When quite hard, let cool, remove the mass with an old file, and pulverize it. Boil up with 500 cubic centimeters of water and pass a stream of carbon dioxide into the hot solution for three-quarters of an hour. The color of the solution changes from green to violet and a brown precipitate is thrown down. The excess of potassic hydrate is converted into potassic carbonate. Allow the liquid to stand a little while; then decant the supernatant liquid through an asbestos filter, with Buchner funnel; finally, filter off the precipitate, and evaporate the filtrate rapidly over the free flame in an evaporating dish till crystals begin to appear; then let cool, filter with suction, and dry in a dessicator over sulphuric acid. Evaporate the filtrate for another crop of crystals.

At all times during the process be very careful to keep organic matter out of the reaction mixture.

Reactions:—



Experiment:—

(a) Dissolve a crystal of KMnO_4 in a quarter of a test tube of tap water and boil. What occurs? (Tap water contains organic matter.)

(b) Dissolve a crystal of KMnO_4 in a quarter of a test tube of water, add 10 drops of dilute H_2SO_4 , and pour into FeSO_4 solution. What happens?

INORGANIC PREPARATION XIV.

NICKEL NITRATE $\text{Ni}(\text{NO}_3)_2 \cdot 6 \text{ H}_2\text{O}$.

In an evaporating dish under the hood place 20 grams of metallic nickel and add concentrated nitric acid, a few cubic centimeters at a time, until the solution of the metal is complete. Avoid much of an excess of acid and when the chemical action slows down, heat the dish with the Bunsen flame. When the metal has dissolved, add

two volumes of water and pass H_2S into the solution until no more precipitate forms. (This throws down the sulphides of the impurities of copper, arsenic, lead, bismuth, etc.) Filter and evaporate to crystallization, remembering that slow cooling is necessary for the formation of large crystals. Dry the crystals in a dessicator over concentrated sulphuric acid.

In a dry test-tube heat a crystal of nickel nitrate.

INORGANIC PREPARATION XV.

MERCURIC OXIDE HgO .

In an evaporating dish under the hood put 15 grams of mercury, and treat with strong nitric acid in small portions until the metal is entirely dissolved. Pour the solution into a beaker and add a strong solution of potassic hydrate (made by dissolving one part of KOH sticks in four parts of water) to complete precipitation. The yellow precipitate of HgO is allowed to settle; then is washed four times with water by decantation. Filter on cloth and transfer to a crystallizing dish and heat in the air bath to 300° for one hour. *Do this under the hood*, and call the attention of the instructor to the apparatus before beginning the heating. The heating changes the yellow modification of HgO into the red.

Heat a small quantity of HgO in a test tube with the free flame.

Complete the reactions:—



INORGANIC PREPARATION XVI.

PHOSPHORUS TRICHLORIDE PCl_3 .

(Graebe, *Chemisches Central-blatt*, 72-926.)

Consult the instructor before performing this experiment, and show him the apparatus before putting in the PCl_3 .

Phosphorus trichloride, PCl_3 , is a colorless liquid boiling at 74° . It fumes in moist air and is decomposed by water and many organic substances. Always work with it under the hood and avoid inhaling the vapors from it.

To prepare PCl_3 set up the following apparatus. (A diagram will be found on the blackboard.)

A chlorine generator and two wash bottles, such as were used in the AlCl_3 and CrCl_3 experiments, are connected to a glass tube which leads to the bottom of a 500 cubic centimeter flask, which flask also is fitted with a return condenser (Liebig style). In the flask place 10 grams of red phosphorus and cover it with a layer of 10 grams of PCl_3 obtained from the store-room. Now pass a moderately rapid stream of chlorine into the flask until the phosphorus has *nearly* disappeared; but be sure it is not all used up. Stop the stream of chlorine, disconnect the apparatus, attach the flask to a downward condenser (see diagram on blackboard), and distil the PCl_3 into a *dry* weighed flask. Calculate the per cent. yield of PCl_3 from the given weight of red phosphorus.

Reaction: $\text{P} + \text{Cl}_2 =$

Perform the following experiments under the hood:—

Add a drop of PCl_3 to a beaker containing ten cubic centimeters of water and test the solution with litmus paper and with $\text{AgNO}_3 + \text{HNO}_3$ dilute. Put a drop of PCl_3 on a watch-glass under the hood and blow your breath over it.

INORGANIC PREPARATION XVII.

CRYSTALLIZED SILICON Si .

Mix thoroughly 10 grams of magnesium powder with 40 grams of perfectly dry sand and transfer to a test tube. Put the test tube into an iron jacket and heat with the Bunsen flame, moving the latter continuously along the iron so that the whole tube is equally heated. Do this under the hood, and have the tube supported by an iron clamp and pointed towards the hood wall. When the action is over let cool, and break the test tube and throw into a beaker of dilute hydrochloric acid. (This is to get rid of the inflammable magnesium silicide.) Pulverize the product and filter. Dry the residue thoroughly, mix with 10 times its weight of zinc, and heat to fusion for several minutes in a Hessian crucible in the furnace. Cool. Wash off the surface of the zinc with water and then dissolve it in dilute hydrochloric acid, under the hood, whereby the silicon is left undissolved in the form of glistening black crystals. Wash with water, dry, and bottle.

Reaction: $\text{SiO}_2 + \text{Mg} =$

INORGANIC PREPARATION XVIII.

BORON (AMORPHOUS AND CRYSTALLINE).

About 125 grams of borax is melted to a "glass" in a large porcelain crucible, the cover is put on, and it is allowed to cool. While still hot it is finely pulverized in a mortar, and very quickly 50 grams weighed out, mixed with 25 grams of magnesium powder, pressed hard into a Hessian crucible with a pestle, and a layer of fused and powdered borax is put over the surface. Cover the crucible immediately with a tight fitting iron plate. Borax contains usually 10 molecules of water. $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$. The dehydrated salt is very deliquescent, and therefore, when working with it, proceed as rapidly as possible and use warmed vessels.

Place the crucible in the furnace and heat to a red heat for 15 minutes. Cool; pulverize the product and boil with water; then with dilute hydrochloric acid; then with water again (decanting off the liquor each time), and filter. This gives amorphous — rather impure — boron.

To obtain the pure crystalline variety, mix the amorphous boron with five parts of aluminium scrap in a small covered crucible and melt in the furnace. Let cool, dissolve the aluminium with dilute nitric acid, and the boron crystals will be left behind in the form of quadratic prisms (boron diamond). Try to scratch glass with one of these.

INORGANIC PREPARATION XIX.

BORIC ACID H_3BO_3 .

Dissolve 100 grams of powdered borax in 300 cubic centimeters of nearly boiling water and concentrated HCl to strong acid reaction. Allow to stand till perfectly cold. Filter the crystals on the Buchner funnel and wash with a very small amount of water. Redissolve the crystals in the smallest possible amount of hot water and let cool slowly. Filter with the Buchner funnel and dry a day or so between sheets of filter paper.

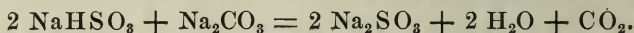
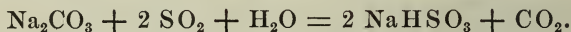
Complete the reaction: $\text{Na}_2\text{B}_4\text{O}_7 + \text{HCl} + \text{H}_2\text{O} =$

INORGANIC PREPARATION XX.

SODIUM SULPHITE $\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$.

Make a concentrated solution of 50 grams of sodium carbonate in hot water, and dilute with two equal parts. Saturate one part (keeping it hot) with sulphur dioxide; then add the second part, mix thoroughly and allow to cool. The sodium sulphite crystallizes out in large crystals. Remove two or three large crystals; dry quickly between sheets of filter paper and preserve in a dry bottle. Place the rest of the crystals back into the filtrate and use in the preparation of sodium thiosulphate.

Reactions:—



This salt is very soluble in cold water. Try a drop of the solution with a piece of turmeric paper. Expose a crystal to the air on a watch glass and note the effect.

PREPARATION OF SULPHUR DIOXIDE SO_2 .

For use in the above experiment the sulphur dioxide may be prepared as follows:—

Place 30 grams of charcoal in a liter flask, which is fitted with a 2-hole rubber stopper; through one hole have a funnel tube reaching nearly to the bottom of the flask; through the other hole have a delivery tube bent at right angles and connected to a gas wash bottle containing 50 cubic centimeters of water. From the wash bottle a tube conveys the gas into the Na_2CO_3 solution. When the apparatus is ready pour, in small portions at a time, 100 grams of concentrated sulphuric acid, warming the flask until the reaction begins. When the Na_2CO_3 solution is saturated with the SO_2 gas, disconnect the delivery tube and stop the evolution of gas by the addition of much water. *Perform this experiment under the hood.*

Complete the reaction: $\text{C} + \text{H}_2\text{SO}_4 = \text{SO}_2 +$

INORGANIC PREPARATIONS. XXI.

SODIUM THIOSULPHATE, $\text{Na}_2\text{S}_2\text{O}_3$.

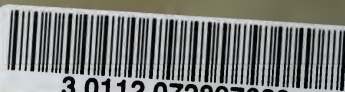
(Prep.)

To the boiling solution of sodium sulphate obtained in the previous preparation and contained in a flask, add flowers of sulphur in small quantities until no more will dissolve. The reaction runs rather slowly. Filter with glazed filter, evaporate the filtrate to a thin syrup, and allow to crystallize. The sodium thiosulphate forms large crystals containing five molecules of water crystallization which are efflorescent.

Heat a crystal in a test tube in the free flame.

Reaction for preparation: $\text{Na}_2\text{SO}_3 + \text{S} = \text{Na}_2\text{S}_2\text{O}_3$.





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